

METHOD FOR THE PRODUCTION OF FEED PELLETS, MIX OF A  
PREMIX AND VITAMIN PREMIX.

5 The invention concerns a method for the production of animal feed pellets with the addition of a premix.

The invention also concerns a method for the mixing of a vitamin premix comprising fat/oil-soluble vitamins.

10 Finally, the invention comprises a vitamin premix comprising soluble vitamins.

15 It is known from EP-A-682874 to add vitamins to animal feeds, said vitamins existing in a fat or oil solution, by which product and by which method there should arise a smaller risk of the decomposition of the vitamin admixture. However, the publication does not disclose a method for the addition of the total physiological vitamin requirement which is necessary in connection with animal feeds, and the publication does not either state any method whereby this admixture of vitamins takes place under conditions  
20 where damage and decomposition of the individual vitamins, whether both fat and water soluble, is minimised to the greatest possible degree.

25 It is the object of the present invention to provide a method whereby the above-mentioned problems are overcome, and whereby the percentage of recovery for the added vitamins is very high, whereby overdosing is avoided, and whereby it is possible to effect a spraying of liquid premixes on the feed pellets, so that investments in larger plants for the handling of premixes following the dry admixture method is made superfluous.

30 It is also the object by this method to ensure that the processing and storage stability is optimised, so that the least possible decomposition of

the vitamins occurs after these have been sprayed on to the industrially-produced feeds.

5 This object is achieved with a method of the kind disclosed in the preamble, and also where the premix is a vitamin premix comprising fat/oil and water-soluble vitamins, where the surface of the feed pellets is sprayed with the vitamin premix, and where the feed pellets have been subjected to cooling- before the spraying.

10 By carrying out a spraying of the feed pellets when these are produced, and by also carrying out a suitable cooling to a maximum of 50°C and an optimum temperature level of 25-35°C, it is possible to effect a subsequent spraying of the surface of the pellets with the vitamin premix, which comprises both fat/oil- and water-soluble vitamins, whereby it is ensured that  
15 the feed pellets receive an optimum content of vitamins for the relevant species of animals, whether these be pigs or chickens etc. By this method, a far higher recovery of the added vitamins is achieved than with a corresponding dosing via a dried vitamin premix.

20 The substantially improved recovery percentage for the vitamins after spraying is of importance for the animals' vitamin supply, and is thus also significant in relation to the feed control authorities. Consequently, it is important that the dosing of vitamins by liquid spraying can be reduced without reducing the animals' supply of added vitamins.

25 By providing a method according to the invention and as further disclosed in claim 2, it is achieved that the feed pellets are stored at a relatively low concentration of oxygen, whereby the decomposition is minimised and the storage stability is optimised. A storage time of 2-6 weeks, which is a  
30 relatively short but nevertheless a realistic storage time, thus results in the vitamin stability and the recovery being surprisingly great.

By providing a method according to the invention and as further disclosed in claim 3, the possibility is established for a regulation of the finish-produced feed pellets with the vitamin premix with which they are sprayed, all depending on whichever animal may be involved. In other words, this means that where the feed is to be used, for example, for chickens, the pellets can be sprayed with vitamins comprising the C vitamin.

By providing a method according to the invention, and as further disclosed in claim 4, a good spraying on the individual pellets is achieved, so that any section of the pellets will give rise to substantially the same amount of concentration of the premix with which they are sprayed.

By providing a method according to the invention and as further disclosed in claim 5, it is achieved that the feed pellets also come to comprise the minerals which may be relevant for the individual animals.

By providing a method according to the invention and as further disclosed in claim 6, the possibility is achieved of adding further nutrients such as lysine, methionine, threonine, leucine and isoleucine.

By providing a method according to the invention and as further disclosed in claim 7, the possibility is achieved for the addition of enzymes such as carbohydrate- and protein-spliced enzymes.

By providing a method according to the invention and as further disclosed in claim 8, a saving is achieved in the amount of added phosphor, and which otherwise means that the selected B vitamin should predominantly be riboflavin and not sodium riboflavin phosphate.

As disclosed in the preamble, the invention also concerns a method for the mixing of the actual vitamin premix which is used during the spraying, and

where the premix also includes water-soluble vitamins, that the water phase comprises propylene glycol and EDTA and nicotinamide, after which there is subsequently added a B<sub>2</sub> vitamin such as riboflavin and sodium hydroxide (NaOH).

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With such a premix comprising the above-mentioned components, the possibility is provided of dissolving the riboflavin, which is important in that riboflavin is not only advantageous from the point of view of cost, but also necessary to use as B<sub>2</sub> vitamin if phytase enzyme is to be added later.

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By providing a method according to the invention and as further disclosed in claim 10, the possibility is provided of being able to add greater amounts of B<sub>2</sub> vitamin such as riboflavin.

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By providing a method according to the invention and as further disclosed in claim 11, a reduction in the pH is achieved, so that the added riboflavin or sodium riboflavin phosphate is not ruined by the high pH which is established by the addition of sodium hydroxide (NaOH).

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By providing a method according to the invention and as further disclosed in claim 12, an expedient composition of the oil phase is achieved.

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By providing a method according to the invention and as further disclosed in claim 13, a homogenous substance of the premix is achieved during stirring.

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By providing a method according to the invention and as further disclosed in claim 14, it is achieved that the amount of phosphate secreted by the animals is reduced. Moreover, the solubility of the riboflavin is ensured due to the presence of urea.

The temperature of 20-30° in the premix can be achieved by the addition of cold water before the addition of the phytase.

5 The invention also concerns a vitamin premix such as that disclosed in claim 15.

The invention will be explained in more detail with reference to the following example:

### LIQUID VITAMINS

Code	Raw material	Weight in grams	
01-A	Vitamin-A-acetate 2.5 mill. IU/g	0.930	Oil phase
02-A	Vitamin-D <sub>3</sub> -oil 4.0 mill. IU/g	0.060	-
03-A	Vitamin-E-oil 97%	39.950	-
04-A	Ethoxyquin	2.000	-
05-A	Bredol 694	50.000	-
01-B	Choline chloride 75% solution*	0	Water phase I
02-B	Vitamin-B <sub>12</sub> 2% solution	0.610	-
03-B	Panthenol	5.100	-
04-B	Biotin 2% to liquid vitamins	1.040	-
05-B	Pyridoxine hydrochloride	1.240	-
06-B	Sodium riboflavin phosphate	0.590	-
07-B	Sodium hydroxide solution 34° Bé	2.600	
08-B	Riboflavin 97%	0.590	-
09-B	Hydrochloric acid 10%	2.800	-
09-B	Folinic acid 100%*	0	-

Code	Raw material	Weight in grams	
10-B	Urea 46 (Carbamide)	50.000	-
11-B	Nicotinamide (=niacinamide)	9.050	-
12-B	Potassium sorbate (preservative agent)	2.000	-
13-B	Tetracemindinsodium (complex binder)	2.000	-
14-B	Propylene glycol (dissolution- promoting and preservative agent)	100.000	-
15-B	Water	666.550	-
01-C	Phytase solution 5000 FTU/g	60.000	Water phase II
02-C	Thiamine hydrochloride	0.990	-
03-C	Menadione sodium bisulphate 100%	1.900	-
	Total – grams:	1000.000	

\* relevant for poultry.

- 5 Before the 07 base is added, the pH is approx. 6, after which the base addition results in a pH increase to approx. 10-11, which is necessary in order for the riboflavin (08) to be dissolved. Component 09 is then added to effect a reduction of the pH to around 8, in that the substance 08 is ruined by a pH which is too high. By carrying out a mixing as described, it is achieved that all vitamins are present in the desired concentrations, and without
- 10 destruction of these taking place.

Generally, the carbamide weight percentage interval of the total amount will lie in the interval 2-10%, mainly around 5%.

If the sodium riboflavin phosphate is omitted, the necessary amount of carbamide in the example will increase to 50-60 grams in order to ensure the solubility of the riboflavin.

- 5 The solution itself consisting of both water-soluble as well as oil-soluble vitamins is subsequently sprayed on the feed pellets for animals. This spraying can be effected before the pellets are stored in silos, or spraying can be effected when the pellets are loaded into transport containers. Both solutions enable a differentiation to be made between the different animals, e.g. pigs vs. chickens, and differentiation in accordance with customer requirements.

10 It has shown surprisingly that when this spraying of the vitamins on the pellets is carried out, no destruction or precipitation of the vitamins occurs, which means that the pellets can be considered to be particularly stable. The reason why no destruction occurs can possibly be that the pellets are stored in silos and containers in which there is a low percentage of oxygen.

#### 20 Production principle

25 The final preparation consists of an oil phase comprising the so-called fat-soluble vitamins mixed up with an adjusted amount of a suitable solubility product – here, use is made of Bredol 694. This mixture, preheated to 55-65°C, is poured/pumped in an adjusted flow down into the water phase containing the water-soluble components and with a temperature set at 40-45°C. The distribution of the oil phase with subsequent dissolution in the water phase, the actual solubilising process, must take place under constant stirring and at a suitably calm pace.

### The mixing procedure

5     The oil phase (code A): The auxiliary material 05 is added one at a time and in the given sequence, the vitamins 01, 02 and 03 and the antioxidant 04. The components are preheated in order to achieve a suitable consistency for handling. Mixing is effected to uniformity. Hereafter, the mixture will continue to be homogeneous.

10     The water phase I (code B): Measured amounts of water 15 (preheated to approx. 45°C) are mixed with 14, in which 13, 12, 11 and 10 are dissolved in this order. Thereafter, 08 is added to the mixture (insoluble in water at this process stage). When the material appears to be distributed uniformly in the water phase, the base 07 is added. Upon conclusion of the dissolution of 08, the acid 09 is added and thereafter 06, 05, 04, 03 and 02 one  
15     at a time and in this order. The product appears as a slightly opalescent liquid.

20     Solubilising phase (A  $\Rightarrow$  B): The oil phase A is now pumped in a thin and continuous stream down into the water phase I, which is held in constant movement by the slowly-moving stirring unit. Mixing is effected to uniformity.

25     Completion (C  $\Rightarrow$  (A + B)) Phytase (01), performed under water phase II, is mixed in, after which 02 and finally 03 are dissolved. The preparation is now ready for use.

In certain cases, the components 02 and 03 can be added in water phase I.



The phytase must be included as a part of the vitamin mixture. This gives rise to the undesired effect that the content of the otherwise water-soluble sodium riboflavin phosphate, when it exists above a certain concentration, is precipitated as riboflavin. The process, which is irreversible under the conditions determined here, will render the finished product useless. The speed at which the precipitation takes place depends first and foremost on the concentration of said components.

Urea (carbamide) is used here as a solubility-promoting material for riboflavin and is optimised for the respective formulation (model). With dosing of riboflavin below a certain level, it is not always necessary to add urea, in that it can be sufficient here with the declared amount of nicotinamide (= niacinamide). It is otherwise well-known that this vitamin of the B-group similarly possesses solubility-promoting characteristics. When the use of urea is found in the actual context, it will form part with 2-10% related to weight of the total product.

If folinic acid is included in a formulation, this is handled in the same way as riboflavin. The material is normally insoluble in water with neutral pH, but can be dissolved in basic solutions where, however, it is chemically unstable. Both nicotinamide and urea serve to promote solubility.

Urea has been selected for the reason that it exits in an acceptable degree of purity, and at the same time it is found to be reasonably cheap for the purpose. Moreover, no negative stability has been shown with regard to the remaining active material contents, and in the stated concentrations it must be considered to be harmless for internal use.

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### Liquid vitamin mixture, sprayed on after pelleting.

5 In the following discussion of the results from the spraying of liquid vitamin premix on cooled feed pellets, the tests with broilers have been omitted, the reason being that the liquid vitamin premix in these tests was introduced via drinking water. However, technically and from the point of view of nutrition, there is nothing to prevent the spraying of liquid vitamin premixes on poultry feeds and all other pelleted types of feeds.

10 Preliminary investigations gave rise to a great deal of conjecture concerning the shown vitamin loss in the production of animal feeds, and therefore it was decided to examine whether feed pellets could be sprayed with liquid vitamin premixes after cooling with a better recovery result – and herewith a better correlation between the added vitamin and the vitamin supplied to  
15 the animals. Similarly, it was decided to carry out production tests with decreasing dosage of liquid vitamin premix compared with dry vitamin premix, the object being to determine a possible minimum dosage where the animals reacted via their feedstuff utilisation or growth.

20 In the trial, the highest dosing of liquid premix corresponded to the dosing of dry vitamin premix to the control feed (index 100 corresponding to standard). The dosing of vitamin to the 4 remaining trials mixtures was reduced to following index: 85, 70, 55 and 40. The only difference in the control and trial feed was the premix type and the method of addition.

25 All mixtures were dosed with 2 kg. per ton feedstuff.

Table 1. Feeds for porkers. Recovery percentages for vitamins in premixes and pellets at production.

	Dry premi x	Feed pellets with dry premix	Liquid premix	Feed pellets with liquid premix
Vitamin-A	79	58	83	77
Vitamin-E	92	68	103	85
Vitamin-K <sub>3</sub>	94	4	82	32
Vitamin-B <sub>1</sub>	60	39	70	82
Vitamin-B <sub>2</sub>	93	74/12	101	226/72
Vitamin-B <sub>6</sub>	67	33	91	71
Vitamin-B <sub>12</sub>	42	18	82	63
Niacin	91	48	97	90
Pantothenic acid	81	57	98	-
Biotin	94	40/-19	95	132/53

- 5 From Table 1 it will be seen that the recovery percentages for vitamins sprayed on the surface of cooled feed pellets are very much higher than for dry vitamins which have gone through the feed production process. However, it must also be ascertained that the recovery percentages lie below the sprayed-on amounts, with great variation from vitamin to vitamin. The investigation does not clarify the question of whether there is an actual vitamin loss due to chemical reactions upon the contact of the vitamins with the surface of the pellets, or whether an analysis problem is involved.

- 15 The vitamins K<sub>3</sub>, B<sub>12</sub> and biotin had very low recovery percentages after dry addition. With liquid spraying, K<sub>3</sub> and biotin still have low recovery percentages, but nevertheless the recovery is 20 to 25 percentage points higher. For vitamin B<sub>12</sub>, the recovery is 30 to 40 percentage points higher.

For vitamin B<sub>1</sub>, the results indicate a recovery of around 80 percent, which is 35 to 40 percentage points higher than with dry vitamins. The spraying-on of vitamin B<sub>2</sub>, B<sub>6</sub> and niacin shows recovery percentages which are 30 to 40 percentage points higher than with the use of dry vitamins. The vitamins A and E have always attracted the greatest interest in analytical post-control of the vitamin contents of the feed mixture – the average results of the present tests show approximately 20 percentage points better recovery for sprayed vitamin A and approx. 15 percentage points for vitamin E.

The improved recovery percentages for the vitamins after spraying are naturally of importance for the animals' vitamin supply, and they are not without significance in relation to the feed control authorities. However, the most important acknowledgement is that the vitamin dosing by liquid spraying can be reduced without reducing the animals' supply of the added vitamins.

#### The storage stability

The recovery percentages for vitamins added via dry vitamin premixes or sprayed on cooled pellets immediately after production can not stand alone – the storage durability of the vitamins is of great importance both for the feed industry and the animal breeder.

To follow up on this, samples were taken of a number of the tested feed portions after storage for 2, 4 and 6 weeks in a feed silo. The relatively short storage time is considered to be appropriate for most of the feed mixtures used in modern animal breeding.

Table 2: Storage stability of vitamins in feed pellets. Percentage recovery after pelleting and 6 weeks storage in relation to meal product (natural vitamins) or declared additives (dry and liquid premix).

Index 100 = standard. Average of 4 productions.

	Feed pellets		Feed pellets with dry pre-mix added, Index 100		Feed pellets with liquid premix added	
	Natural vitamins				Index 100	
	Production	6 weeks	Production	6 weeks	Production	6 weeks
Vitamin-A, I.u./g	280/100	280/100	58	59	79	65
Vitamin-E, mg/kg	112	101	68	65	79	96
Vitamin-K <sub>3</sub> , mg/kg	120	44	4	0/23	48	4
Vitamin-B <sub>1</sub> , mg/kg	91	108	51	36	84	25
Vitamin-B <sub>2</sub> , mg/kg	114	117	43	38	67	95
Vitamin-B <sub>6</sub> , mg/kg	130	88	33	51	82	88
Vitamin-B <sub>12</sub> , mg/kg	142	98	17	30	76	50
Niacin, mg/kg	102	103	48	55	84	67
Pantothenic acid mg/kg	92	88	57	68	-	-
Biotin, mg/kg	94	101	20	25	50	75
					13/131	0

Table 2 shows recovery percentages for vitamins at production and after storage for 6 weeks. For feed pellets which only contain natural vitamins, the recovery in pellets is set in relation to the contents in the meal product. For the remaining mixtures, the recovery is expressed in relation to the guaranteed addition. Recovery after 2 and 4 weeks' storage is omitted out  
5 of regard for space, and because they do not influence the general tendency.

10 In general it can be ascertained that all naturally occurring vitamins would appear to have a greater storage stability.

Vitamin A shows a slight storage loss after spraying, but the loss is considerably less than expected. For the vitamins E, B<sub>2</sub> and B<sub>6</sub>, there is no systematic storage loss, and pantothenic acid added via dry premix and in  
15 natural form appears to be very stable. Pantenol applied via liquid premix can also be assumed to be a stable vitamin. The vitamin K<sub>3</sub> retained after liquid application is quickly reduced during storage and it approaches 0 at 4 weeks' storage, but the amounts shown were found upon analysis after 6 weeks. For vitamin B<sub>1</sub> there is great storage stability for natural vitamin.  
20 During the storage period, the high recovery level after spraying falls down to the same level as found with dry vitamin addition. For vitamin B<sub>12</sub>, great difference is seen between the tests, both with regard to level and tendency. However, the amount of vitamin remaining after storage for 6 weeks will be considerably higher after liquid spraying than after dry addition. The  
25 result for niacin shows a tendency towards greater loss with liquid spraying than with dry addition, while the result for biotin hardly permits itself to be interpreted due to the great variation in data. A guarded interpretation could be that there is 20 to 30 percent remaining after 6 weeks regardless of the method of application.

The general result of the storage test is that the storage stability remains the same regardless of the method of application. The exceptions are vitamin K<sub>3</sub>, B<sub>1</sub> and perhaps B<sub>12</sub>, which after liquid spraying shows some decrease. However, the contents of these vitamins at the start of the storage test were considerably higher after liquid spraying than with dry addition. If there are to be reasonable amounts of vitamin K<sub>3</sub> and B<sub>1</sub> in the feed after liquid spraying, the feed must be used within 2 to 4 weeks after production.

The tests carried out show a surprisingly great process loss of all vitamins added to the feed via dry vitamin premix. The lacking vitamin recovery starts already after admixture with the meal product. A number of vitamins lose further activity during expansion, and there is a tendency towards continued loss during the subsequent pelleting. In feed pellets, the average recovery for added vitamin E and pantothenic acid is highest with approx. 70 percent, while the recovery percentages for the remaining vitamins lie a good 50 to approx. 25 percent down. Vitamin K<sub>3</sub> is particular in that it more or less disappears.

The average recovery percentages for vitamins sprayed on feed pellets after cooling lies from 15 to 60 percentage points higher than for vitamins added in the form of dry vitamin premix (table 1).

Since at the same time the storage stability for the vitamins with up to 4 weeks' storage of the feed mixtures is generally the same regardless of the method of application, the animals are provided with considerably higher amounts of vitamin at the same vitamin dosing to the feed when the vitamins are sprayed on as liquid premix.

The content of natural B vitamins proves to be extremely stable during processing, and even with a tendency towards increasing content during the feed production process. With the content level taken into con-

sideration, it is not curious that a feed mixture seldom or never falls at the post-control for the content of B vitamins. The same applies to a considerable extent regarding vitamin E. Vitamin A and especially vitamin K<sub>3</sub> do not have this natural back-up, and since both vitamins in synthetic form are somewhat or very sensitive to processing, it is obvious that post-control here will reveal many failures.

The recovery percentages in pellets for vitamins added via dry vitamin pre-mixes show that there is a very poor correlation between the declared vitamin content (added) in the feed mixtures and that amount which reaches the animals. The relationship is considerably better when the pellets are sprayed with vitamins. In light of this, it was natural to carry out an investigation into whether the animals in production tests reacted to reduced vitamin addition via liquid premix sprayed on the cooled feed pellets.

The results of the production tests showed that porkers did not react to a reduction of vitamin allocation via liquid spraying of 60 percent of the prescribed dry vitamin addition.

The tests performed show that a considerably higher percentage of the added vitamins reach the animals when the vitamin premix is sprayed on the finished feed or dosed via the drinking water. Consequently, due to the elimination of the process loss, the animals' vitamin requirements can be covered with a smaller addition of vitamins after pelleting.

The concept involves the addition of phytase enzyme to expanded/pelleted poultry feeds and pig feeds. When the enzyme is sprayed on cooled feed pellets together with the vitamin premix, the full enzyme activity is retained, which gives rise to the release of 60 to 70 percent of the organically-bound phosphor in vegetable feed raw materials. Furthermore, smaller amounts of amino acids, carbohydrates and micro-minerals are released. As a con-



- 5 The micro-minerals in a liquid micro-mineral mixture are 100 percent dissolved, the result being that the accessibility to and the absorption by the animals is greater than with the use of traditional salts. As a consequence, the dosing of micro-minerals to industrially-produced feeds can be reduced, with subsequently less discharge to the environment.

The new products can be delivered in packaging which can be handled by truck, and the products are conveyed from the packaging to the spraying equipment in the feed mill through closed piping systems. This results in considerable advantages for the animal feed industry, both with regard to

15 handling as well as the working environment.

**REPORT OF THE**